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### (54) Multilayer stretch/shrink film

Mehrschichtiger streckbarer Schrumpfilm  
Film thermorétractable et étirable multicouche

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US-A- 4 985 188

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## Description

## Field of the Invention

This invention relates to an improved biaxially oriented heat shrinkable multilayer film, a hermetically sealed and evacuated trayed food package wherein the film is used as a stretched heat shrunk overwrap and a method for sealing and wrapping such packages.

## Background of the Invention

For many years, food products such as fresh poultry were shipped in bulk from the food processors to supermarkets where the bulk pieces were divided into small quantities and repackaged for retailing. For example, fresh poultry was cut up, placed on cardboard or plastic trays, and covered with stretch film secured to the tray by tack welding.

For improved efficiency the current trend is to perform the retail packaging operation in the food processing plant and ship the small retail packages from such plants to the retailers. There is also a trend towards evacuating and hermetically sealing the food retail packages in the central food processing plants, due to the longer storage time between retail packaging and consumer use. Such packaging increases the shelf life of the food packages. Also, there is a need for increased abuse resistant food retail packages because of the more frequent handling, impact and abrasion inherent in the above-described food processing plant retail packaging system.

This retail packaging can be accomplished in a variety of ways. A number of systems are available to food processors for wrapping and heat sealing poultry-containing trays with plastic overwrap film. One packaging system that uses a continuous belt sealer is the Ossid™ 500, produced by the Ossid Corporation. (Ossid™ is a trademark of the Ossid Corporation, North Carolina, USA.) Generally, a poultry-containing tray is moved by a series of conveyors and rolls while a film is fed from a dispenser and mechanically pulled over the tray top. The film edges are wrapped around the tray, pressed against the tray bottom, overlapped and sealed against it. Finally, the tray is moved forward and the opposite ends cut. In the Ossid™ 500 system both cut portions of the film, the flaps, are pulled under the tray and shaped under the tray.

Sealing is done with heat and pressure. In impulse sealing systems, the contact temperature ranges from 400 to 800°F

$$(\alpha^{\circ}\text{F} = \frac{(\alpha - 32)}{1.8}^{\circ}\text{C})$$

with a high applied pressure due to the narrow sealing surface. In a bar heating sealing system, the temperature ranges from 250 to 400°F with a similar pressure as above. The heat contact time is longer than with the impulse system. The Ossid 500 employs a continuous belt sealer that consists of a release belt that is drawn over a heated platen. The resultant platen seal has characteristics that vary significantly from other commercial sealing processes. In the platen heating-sealing system, the temperature is at 250 to 400°F, using a low pressure of 0.1 to 2.0 psi (1 psi = 6.89 kPa), with the heat contact time being 2 to 4 seconds.

The wrapped tray is passed to the belt or platen which applies heat that bonds the film layers together and, upon cooling, the seal is fixed. The heat bonded film is then immediately cooled to a temperature below 200°F.

The machinability of the film, or the way that it operates on the machine, is an important characteristic of the film used.

The most commonly used film in the trayed food overwrap market is polyvinyl chloride (PVC). This thermoplastic polymer has been used because of satisfactory elongation and elastic memory. But in some instances the package becomes leaky during shipment and thereby unacceptable to the customer. This is because the PVC material in overwrapped trays is tack welded to itself and not hermetically sealed. This problem cannot be solved by hermetically sealing because of a very narrow sealing temperature range between which the seal is effective at the low temperature end and "burn through" or melting of the PVC at the high temperature end. This range is so limited as to be impractical with many commercially employed heat sealing systems.

Another limitation of PVC tray overwrap material is its poor resistance to physical abuse. The PVC material tends to tear along the edges of the overwrapped tray if rubbed during transit by another tray or an enclosing carton.

One characteristic of PVC is that although it contracts, it is not generally "heat-shrinkable", i.e., it is a material that tends not to return to its original unstretched (unextended) dimension when heated to its softening point. The terms "orientation" or "oriented" are used to describe the manufacture of heat-shrinkable films, wherein resin material is heated to its flow or melting point and extruded through a die into either tubular or sheet form. After cooling, the relatively thick extrudate is reheated to a temperature range suitable to orient or align the crystallites and/or molecules of the material.

The orientation temperature range for a given material or materials is understood by those skilled in the art to be in a range which revises the intermolecular configuration of the material by physical alignment of the crystallites and/or molecules of the material to improve certain mechanical properties of the film such as shrink tension as, for example, measured in accordance with ASTM D-2838-81. When the stretching force is applied in one direction, uniaxial orientation results. When the stretching force is simultaneously applied in two directions, biaxial orientation results.

In view of the limitations of PVC as a stretch film food tray overwrap material, there have been prior efforts to identify a heat-shrinkable thermoplastic film having an improved combination of elongation, elastic memory, heat sealability and puncture resistance. However, most heat-shrinkable thermoplastic film packaging materials suitable for food contact have relatively poor elasticity or elastic memory. Thus, when a food wrapped in such a material shrinks from moisture loss, the film does not shrink, resulting in a loose package that is unacceptable.

One PVC replacement film for use as a trayed food overwrap material is described in U.S. Patents 5,272,016 and 5,279,872, issued to D.J. Ralph ("Ralph"). The Ralph film is the biaxially oriented heat-shrinkable multilayer stretch type, comprising at least a first outer layer, a second outer layer, and a core layer between the first and second outer layers. The outer layers each comprise a blend of between about 20 and about 35 wt.% ("wt.%") ethylene  $\alpha$ -olefin plastomer copolymer of density below about 0.90 g/cm<sup>3</sup>, and between about 65 and about 80 wt.% very low density polyethylene ("VLDPE"). The core layer comprises ethylene  $\alpha$ -olefin copolymer having a higher melting point than the melting point(s) of either of the first or second outer layers. By way of example, the core layer may be polypropylene or a polyolefin. Embodiments of the latter include VLDPE, linear low density polyethylene ("LLDPE"), and blends of two different density VLDPE's or VLDPE and LLDPE.

The Ralph-type film in nonirradiated form has been demonstrated to be suitable as a PVC replacement for food tray overwrapping and sealing by the impulse and bar heating-sealing systems. However, it has substantial limitations when used in platen sealing systems.

When the heat seal between the film layers forming the end flaps is not complete, the package is defective. Incomplete seals represent potential air/fluid leaks and loss of food quality and/or desired appearance in the retail market. As seen from the above, the number of overlapping film layers to be sealed together may vary substantially, generally from six to at least twenty. This means that the required maximum seal temperature must be relatively high, because as the number of film layers increases, the temperature at a given residence time required for a complete seal increases. However, "burn through" places an upper limit on the sealing temperature. Burn through means the temperature at which any hole or penetration of the film occurs, resulting from melting and/or shrinking of the film during sealing.

When the Ralph-type film with a 100% VLDPE core layer was tested for use on platen sealed poultry-containing trays, the sealing range was too narrow as complete sealing could not be obtained without burn through. In an attempt to overcome this problem, LLDPE was added to the core layer making the core a VLDPE-LLDPE blend. This formulation provided a broad enough platen seal range on an Ossid 500 system under ideal conditions, but under production conditions the sealing range was still too narrow. To broaden the heat sealing range of the VLDPE-LLDPE blend core layer Ralph-type film, the film was irradiated at a dose of 8 MR after biaxial orientation.

Although irradiation of the VLDPE-LLDPE blend core layer Ralph-type film provided the needed wide heat sealing range, a new and unexpected problem developed. The film had poor machinability. The slide characteristics of the film were such that the film could not be smoothly transported through and over the various belts and rollers which are essential to film movement through the wrapping machine. At the same time, it should be recognized that the film cannot be so frictionless that it cannot be secured by the clamps as, for example, chain grips, which grab the film edges and pull it taut over the top of the tray.

During testing, another facet of the machinability problem with the irradiated Ralph-type film, flap pull back, was discovered. Flap pull back refers to the tendency for the folded under flap to be pulled away from the tray bottom surface by movement across the rollers prior to entering the platen sealer-cooler. This problem appears to be related to the frictional properties of the film, possibly the ratio of the friction between the flap folding rollers on the platen sealing-cooling system and the film-to-film slip.

An improved polyolefin-type heat-shrinkable oriented stretch film suitable for use as a platen heat sealed overwrap material for a food tray with a wide sealing range and good machinability is needed. This film should also be characterized by good elongation, good elastic memory, puncture resistance and abrasion resistance.

An improved method for wrapping and platen-sealing a polyolefin stretch-shrink film as an overwrap on a food-containing tray is also needed.

Also needed is an improved evacuated hermetically sealed food-containing tray overwrapped by a heat shrunk polyolefin film.

#### Brief Description of the Invention

An improved polyolefin-type heat shrinkable oriented stretch film suitable for use as a overwrap material for a food tray that is platen heat sealed on an automated system has been invented. This film is characterized by good elonga-

tion, good elastic memory, puncture resistance and abrasion resistance. It also has a wide sealing range and good machinability, especially in the platen-type heat sealing system.

One aspect of this invention relates to a crosslinked biaxially oriented heat shrinkable multilayer stretch film comprising at least one outer layer, a second outer layer and a core layer between the first and second outer layers. The outer layers each comprise a two component polyethylene blend of between 25 and 75 wt.% VLDPE of density between 0.900 and 0.914 g/cm<sup>3</sup>, and between 25 and 75 wt.% LLDPE of melt index below 3.5 g/10 minutes and density between 0.917 and 0.925 g/cm<sup>3</sup>. The LLDPE comprises less than 35 wt.% of the total film weight.

The core layer of this film comprises a three component blend of between 40 and 75 wt.% first VLDPE having a density between 0.905 and 0.914 g/cm<sup>3</sup>, between 10 and 35 wt.% second VLDPE of density between 0.900 and 0.905 g/cm<sup>3</sup>, and between 15 and 35 wt.% ethylene  $\alpha$ -olefin plastomer copolymer with a density below 0.900 g/cm<sup>3</sup>. In this film, the core layer has a lower composite melting point than do the outer layers.

The film of this invention has biaxial heat shrink properties and can be crosslinked such that when subjected to the Platen Test (described below) on a plate heated to a contact surface temperature range between 280°F and 400°F, for a contact time of between 2 and 4 seconds, the film heat seals and does not burn through. Heat shrinkable means that the film has at least 10% free shrink at 90°C measured in both the machine and transverse directions in accordance with ASTM D-2732.

Another aspect of the invention relates to a hermetically sealed and evacuated food package comprising a tray with a bottom section surrounded by upwardly extending side and end walls, perishable food supported on the upper surface of the bottom section, and a stretched heat shrunk film extended over the food, the upper edges of the side walls and at least part of the lower surface of the tray bottom section and heat sealed to itself in flattened relationship against the lower surface so as to form with the tray a hermetically sealed enclosure for the food. In this aspect of the invention, the improvement is the biaxially oriented multilayer composition as the stretched heat shrunk film described in the paragraphs above.

Still another aspect of this invention relates to a process for platen heat sealing the overlying folded ends of multiple contiguous layers of a stretched thermoplastic film covering a food-containing tray having side and end walls upwardly extending from a bottom section wherein the multiple layers of film are folded over the tray end walls, pressed against the lower surface of the tray bottom section and interlayer heat bonded together to form a hermetically sealed and evacuated food package. The improvement comprises using as the thermoplastic film the above described at least three layer film article, providing a flat metal plate with a release surface as the platen surface, heating the upper surface of said plate to temperature between 250 and 400°F, contacting pressed folded end portions of the stretched film cover with the heated metal plate for a period of between 2 and 4 seconds so as to bond the contiguous film layers of said folded end portions together without burn through of said film, and thereafter immediately cooling the heat bonded end portions of said film to temperature below about 200°F.

#### Brief Description of the Drawings

Figure 1 is a cross-sectional view of the flap folding mechanism.

Figure 2 is a cross-sectional elevation view of a sealer-cooler system.

#### Detailed Description of the Invention

In the food industry and, in particular in the poultry industry, small quantities of poultry are cut up and packaged in trays which are overwrapped with film. This packaging allows retail sized quantities of food to be prepackaged by the food plant and shipped to retail outlets in a condition acceptable to both the health department authorities, the retailer and the final consumer.

The various film-shaping steps in a representative method for covering a food-containing open tray with a stretch-shrink film using the hot platen sealing system is described below. Each of the following steps is performed automatically on an overwrap system, in particular, the Ossid™ 500, starting with a tray of food or, particularly, poultry.

Food containing open trays are longitudinally spaced and moved forward on a feed conveyor belt. The feed belt is supported at opposite ends by rolls and moves continuously to carry trays to a position beneath a film roll dispensing system positioned above the conveyor belt. When a sensor detects the presence of the food containing tray beneath the feed roll system, film is then dispensed from the supply roll through a series of guide and tensioning rolls as will be understood by those skilled in the art. The dispensed and tensioned film is seized and stretched by a series of clamps as, for example, chain grips longitudinally aligned on opposite sides of the dispensed film edges at progressively increasing distances from the longitudinal centerline of the system in the direction of the film movement. Downwardly moving clamps grab the films out edges and progressively pull the film tautly over the top edges of tray sides. There are two points in the chain gripping system where problems can occur with the wrong type of film: the film can be either too slippery for the clamps to correctly grasp it, or not slippery enough so that the film does not dispense easily and

therefore jams up in the dispensing system.

The clamps are activated by cams at each end of chain grip belt to grab and release the tensioned film at respective opposite ends of the belt. Upon release from the grip clamps, the film is pulled under the tray by plates and center rollers. The two edges of the film are overlapped and can be longitudinally sealed together by heated rollers. The tubular film-enclosed tray is driven forward by belt conveyor having flexible, eg., elastomeric contact surfaces including side walls. The transverse distance of these side walls from the longitudinal centerline can be adjusted depending on the tray size and film thickness.

After the tube film longitudinal center seal is completed, the opposite ends are cut by knives. Next, the cut portion of the tube film extending in front of the tray (ie., the front flap) is pulled under the tray by a roller-vacuum system. Flap guides are located on each side of the tray to help shape the front flap so that it does not extend beyond the bottom edges of the tray. At this point in the film packaging system, the front end film has been stretched over and under the tray front end against the tray bottom and towards the tray rear end. The flap folding areas of the machine are also areas where film with incorrect frictional properties will jam up the overwrap process.

As shown in Figure 1, the partially film 5 enclosed food-containing tray 1 then passes over the rear film flap folding conveyor 2 which, for example, may comprise a series of plastic rollers 3 with a leading metal roller 4. The latter is longitudinally spaced from the next forward roller to provide a gap. The trailing flap 6 is pulled with this gap by a mechanically applied vacuum system. The rollers 3, which are moving faster than the tray 1, flatten the trailing flap 6 under the tray 1 to form the food package.

As the rear flap is being folded under, the front flap can be pulled out. If frictional forces are higher between the film and the metal roller of the flap folding area than between the film and itself, the front flap is pulled out. If the front flap is not completely sealed, the whole package must be reworked.

The stretch wrapped food package is now moved to the sealer system.

As seen in Figure 2, the bottom surfaces of foam pillows 12 in the sealer system are in direct contact with the wrapped food package 1 to apply uniform pressure to insure efficient heat transfer by solid conduction from the heated metal platen 13 to the film end folds pressed against the bottom outer surface of the tray 1. Heat is applied by a heating means, eg., electric resistance or circulating fluid. A representative temperature range for the heated platen top surface in the practice of this invention is 300-400°F. Based on a belt sealer conveyor 14 speed of 0.254 m/s (50 ft/minute) and a 40" (1" = 25.4 mm) long heating platen 13, the tray-platen contact time is 2 - 4 seconds.

After heat sealing the flap ends to themselves as supported by the food package bottom surface, the food package is conveyed to a cooling platen 15 that is cooled. The top surface of the platen may be cooled to about 55-65°F and is in contact with the tray 1 for a sufficient amount of time to cool the heated film below 200°F.

If the film used to overwrap the food trays has the wrong machinability qualities, the trays will not be usable. The film must be slippery enough to slide through the overwrap machine without jamming, but must be able to be held tautly in place when being stretched over the food and maintain this tautness until sealed. It must allow the trays to continue their forward movement on the conveyor belt once the flaps have been folded under. And finally, it must be able to be completely heat sealed without burning through the layers of film.

The polyolefin multilayer film of this invention requires at least three layers, two outer layers and a core layer between the outer layers, has biaxial heat shrink properties and is crosslinked. Stretch recovery and abrasion resistance of the film meets the requirements of the food packaging industry. It exhibits good machinability, overcomes the problems associated with an irradiated Ralph film, has adequate slip properties, is resistant to burn through and has no flap pull back.

The film of this invention is a crosslinked biaxially oriented heat shrinkable multilayer stretch film comprising at least a first outer layer, a second outer layer and a core layer between the first and second outer layers. The outer layers each comprise a two component polyethylene blend of between 25 and 75 wt.% VLDPE of density between 0.900 and 0.914 g/cm<sup>3</sup>, and between 25 and 75 wt.% LLDPE of melt index below 3.5 g/10 minutes and density between 0.917 and 0.925 g/cm<sup>3</sup>. The LLDPE comprises less than 35 wt.% of the total film weight. A preferable range of components is from 30 to 40 wt.% of VLDPE and from 60 to 70 wt.% of LLDPE. Additives can comprise up to 10% of the final formulation.

The core layer of this film comprises a three component blend of between 40 and 75 wt.% first VLDPE having a density between 0.905 and 0.914 g/cm<sup>3</sup>, between 10 and 35 wt.% second VLDPE of density between 0.900 and 0.905 g/cm<sup>3</sup>, and between 15 and 35 wt.% ethylene  $\alpha$ -olefin plastomer copolymer with a density below 0.900 g/cm<sup>3</sup>. A preferable range of components is from 60 to 75 wt.% of the first VLDPE, 13 to 20 wt.% of the second VLDPE and 15 to 25 wt.% of the plastomer. Also, the ratio of plastomer to second VLDPE should range from 0.77 to 1.83, preferably from 1.29 to 1.42. Additives can comprise up to 3% of the final formulation of the blend.

Polyolefins are hydrocarbon polymers derived from a simple olefin, such as polyethylene or polypropylene and copolymers of such olefins. Their basic structure is characterized by the chain  $(CH_2CH_2)_n$  and they may be found as a homopolymer or as a copolymer. Polyolefins used in the present invention, unless otherwise noted, are substantially free of halogens, oxygen or other elements apart from carbon and hydrogen, except for incidental amounts, e.g., trace residues of catalysts or process related contaminants of the above.

The outer layers are a two component polyethylene blend of VLDPE and LLDPE, while the core layer is a three component blend of two VLDPE's and a so-called ethylene  $\alpha$ -olefin "plastomer".

One type of polyethylene is known as Linear Low Density Polyethylene ("LLDPE"). Only copolymers of ethylene with  $\alpha$ -olefins are in this group. LLDPEs are presently recognized by those skilled in the art as having densities from 0.917 to 0.940 g/cm<sup>3</sup>. The  $\alpha$ -olefin used is usually 1-butene, 1-hexene or 1-octene. Ziegler-type catalysts are usually employed in their production, although Phillips catalysts are also used to produce LLDPE having densities at the higher end of the range. LLDPEs typically do not have many long branches off the main chain.

Another form of linear polyethylene is Very Low Density Polyethylene ("VLDPE"), also called Ultra Low Density Polyethylene ("ULDPE"). The densities of commercial VLDPEs are recognized by those skilled in the art to range between 0.890 and 0.914 g/cm<sup>3</sup>. VLDPEs comprise copolymers of ethylene with  $\alpha$ -olefins, usually 1-butene, 1-hexene or 1-octene, and in some instances terpolymers, as, for example, of ethylene, 1-butene and 1-octene. As used here, VLDPE also includes terpolymers of ethylene and higher  $\alpha$ -olefin comonomers.

A process for making VLDPE is described in European Patent Document publication number 120,503. As described in EP 120,503, these particular VLDPEs are made by using the traditional Ziegler-Natta heterogeneous catalyst system.

Alternatively, VLDPE and LLDPE may be prepared by a homogeneous metallocene single-site catalyst system which in general produces molecular chains of more uniform lengths with more evenly spaced comonomer. This type of system is described in U.S. Patent 5,183,867, assigned to Exxon Chemical Company ("Exxon") and European Patent Application Publication 0 416 815 A2, assigned to Dow Chemical Company ("Dow").

For example, as described in U.S. Patents 4,640,856 and 4,863,769, VLDPEs are useful in biaxially oriented films which have superior properties to comparable films using LLDPEs. These superior properties include higher shrink, higher tensile strength and greater puncture resistance.

Commercially available ethylene  $\alpha$ -olefin plastomers have densities typically below 0.900 g/cm<sup>3</sup>. Examples of plastomers include the Japanese Mitsui Corporation's ("Mitsui") "Tafmers". As used here, ethylene  $\alpha$ -olefin plastomers include terpolymers of ethylene and higher  $\alpha$ -olefin comonomers. According to US Patent 4,469,753, Tafmers are copolymers of butene-1.

Although it appears that the Tafmer-type plastomers are prepared using the Ziegler-Natta heterogeneous catalyst systems, other ethylene  $\alpha$ -olefin plastomers are manufactured using homogeneous metallocene single-site catalyst systems as previously described.

The following is a general discussion of LLDPE, VLDPE and plastomer properties from several perspectives.

#### Crystallinity

Differential Scanning Calorimetry ("DSC") is commonly used to measure the amount of crystallinity in a plastic sample, while revealing the nature of this crystallinity. As determined in a procedure similar to ASTM D-3418, a DSC is performed by exposing a sample of the plastic to a constant rate of heating, i.e., 50°C per minute in a E.I. DuPont de Nemours Company ("DuPont") brand differential scanning calorimeter. When the temperature of a sample reaches the melting point of a crystalline region, the continued application of heat causes the crystalline fraction to melt, while the sample temperature remains constant. After the crystalline region has melted, the sample temperature once again begins to rise.

DSC measurements were performed on two types of VLDPE's: the Union Carbide Corporations ("Union Carbide") 1137, an ethylene-butene copolymer with 0.906 g/cm<sup>3</sup> density and the Dow Attane™ 4001, an ethylene-octene copolymer with 0.912 g/cm<sup>3</sup> density. The same type measurement was performed with Tafmer A-1085. Each of these ethylene  $\alpha$ -olefin copolymers has some crystallinity but the crystalline nature of the ethylene  $\alpha$ -olefin plastomer and the VLDPE copolymers is entirely different.

The entire crystalline phase of the ethylene  $\alpha$ -olefin Tafmer A-1085 plastomer melts between about 55° and 85°C, and this melting point range is consistent with the crystalline phase being made up of an ordered butene-ethylene copolymer. In contrast, the VLDPE copolymers useful in this invention have at least one crystalline phase with the dominant phase being that of a higher temperature melting point, above about 90°C.

The melting points of representative VLDPE, LLDPE and plastomeric ethylene  $\alpha$ -olefin copolymers useful in the practice of this invention are seen in Table A.

Table A

Ethylene $\alpha$ -Olefin Melting Points (°C)	
Type Compound and Manufacturer	MP (°C)
Dow 2045 LLDPE	121
Exxon 3001 LLDPE	125 <sup>(1)</sup>
Exxon 3201 LLDPE	126 <sup>(1)</sup>
Union Carbide 1137 VLDPE	117
Union Carbide 1085 VLDPE	117
Union Carbide 1092 VLDPE	121
Union Carbide 1063 VLDPE	124
Union Carbide 1064 VLDPE	125
Dow Attane™ 4001 VLDPE	121
Dow Attane™ 4003 VLDPE	107/124(two peaks)
Exxon Exact™ 3025 VLDPE	103 <sup>(1)</sup>
Exxon Exact™ 3034 VLDPE	95 <sup>(1)</sup>
Exxon Exact™ 3033 VLDPE	94 <sup>(1)</sup>
Exxon Exact™ 3027 VLDPE	92 <sup>(1)</sup>
Dow Affinity™ PL 1840 VLDPE	103
Dow Affinity™ PL 1880 VLDPE	100
Mitsui Tafmer A-4085 plastomer	71
Mitsui Tafmer A-4090 plastomer	85
Mitsui Tafmer A-1085 plastomer	71
Exxon Exact™ 4011 plastomer	66 <sup>(1)</sup>

<sup>(1)</sup> Exxon method

Table A demonstrates the substantial difference in melting points of VLDPE and LLDPE as compared to ethylene  $\alpha$ -olefin copolymer plastomers. More particularly, ethylene  $\alpha$ -olefin copolymer plastomers suitable for use in this invention have melting points below 90°C and VLDPE and LLDPE materials suitable for use in this invention have melting points above 90°C. Preferably the plastomers have melting points below 85°C. Preferred VLDPE copolymers for practice in this invention have a crystalline melting point between 92°C and 125°C.

#### Vicat Softening Point

Vicat softening points as performed by ASTM 1525 are reported by the resin manufacturers and are summarized in Table B (Vicat Softening Points).

Table B

Vicat Softening Points (°C)	
Type Compound and Manufacturer	(°C)
Dow 2045 LLDPE	100
Union Carbide 1137 VLDPE	80
Union Carbide 1063 VLDPE	93
Union Carbide 1064 VLDPE	90
Union Carbide 1569 VLDPE	94
Dow Attane™ 4001 VLDPE	95
Dow Attane™ 4003 VLDPE	80
Dow Attane™ 4004 VLDPE	92
Exxon Exact™ 3033 VLDPE	75-83
Mitsui Tafmer A-1085 plastomer	58
Mitsui Tafmer A-4085 plastomer	54
Mitsui Tafmer A-4090 plastomer	65
Mitsui Tafmer A-20090 plastomer	60
Mitsui Tafmer A-0585 plastomer	62
Exxon Exact™ 4011 plastomer	70

Based on the foregoing and for purposes of this invention, LLDPE and VLDPE-type ethylene  $\alpha$ -olefin copolymers useful in the practice of this invention preferably have Vicat softening points of at least 75°C, and most preferably between 78°C and 100°C. Conversely, preferred plastomer-type ethylene  $\alpha$ -olefin copolymers have Vicat softening points below 72°C and most preferably between 50°C and 72°C.

#### Molecular Weight/Size Distribution

Ethylene  $\alpha$ -olefin copolymers may be partially characterized by their weight average molecular weight (Mw) which is determined by multiplying the weight of each chain of a given number of repeat units by the number of such chains and dividing by the total weight of chains. Ethylene  $\alpha$ -olefin copolymers may also be partially characterized by a number average molecular weight (Mn), which is derived from the total weight of polymer molecules divided by the total number of molecules. When both of these are known, they can be used to characterize the shape of the molecular weight distribution curve for the copolymer, i.e., the number of polymer chains in a molecular weight interval as the ordinate and the molecular weight as the abscissa.

A high Mw/Mn connotes a wide distribution of molecular weights whereas a low Mw/Mn connotes a narrow distribution. Mw/Mn can be measured by several different techniques, but the gel permeation chromatography ("GPC") procedure outlined in ASTM D-3593-80 is used.

All of the specific LLDPE copolymers known to applicants as suitable for use in this invention have relatively narrow molecular weight distribution and Mw/Mn values above 3. However, it may be possible to manufacture suitable LLDPE materials having relatively narrow molecular weight distributions and Mw/Mn values below 3. Mw/Mn values for various polyolefins are given in Table C.



Table C

Molecular Weight/Size Distributions	
Type Compound and Manufacturer	Mw/Mn
Dow 2045 LLDPE	4.17 (125,000/30,000)
Union Carbide 1137 VLDPE (0.906 g/cm <sup>3</sup> density, 1.0 MI)	4.9 (125,000/25,700)
Union Carbide 1192 VLDPE (0.912 g/cm <sup>3</sup> density, 0.19 MI)	12.2 (196,900/16,080)
Union Carbide 1096-2 VLDPE (0.912 g/cm <sup>3</sup> density, 0.38 MI)	7.2 (137,000/19,110)
Mitsui 0.907 g/cm <sup>3</sup> den. VLDPE	3.2*
Exxon Exact™ 3033 VLDPE	1.8 (92,000/50,000)
Mitsui Tafmer A-4090 plastomer	2.0*
Mitsui Tafmer A-4085 plastomer	2.35 (108,000/46,000)
Mitsui Tafmer A-1085 plastomer	2.00 (160,000/80,000)
Mitsui Tafmer A-0585 plastomer	2.05 (190,000/92,600)
Exxon Exact™ 4011 plastomer	2 (approximate)

\* Reported by manufacturer in, "Proceedings of Future-Pak '91", page 314. Procedure for measuring Mw/Mn not identified.

#### Tensile Properties

In general, known VLDPE and LLDPE materials useful in the practice of this invention have higher tensile modulus than comparable ethylene  $\alpha$ -olefin plastomers. That is, if the two types of copolymers were prepared from the same comonomer and using the same catalyst system, the VLDPE or LLDPE would have higher crystallinity and density, hence higher tensile modulus.

Mitsui, a manufacturer of both plastomers and VLDPE, reports the following values for Young's Modulus, which is the ratio of stress to strain below the proportional limit of a material, as summarized in Table D.

Table D

Young's Modulus	
Material	kg/cm <sup>2</sup>
Tafmer A-4085*	400
Tafmer A-20090*	600
VLDPE (0.907 density)**	1820

\* "Tafmer", publication of Mitsui Petrochemical Ind, Ltd. page 12

\*\* "Proceedings of Future-Pak '91", page 314

Another difference in the tensile properties of ethylene  $\alpha$ -olefin plastomers, LLDPE and VLDPE materials is that plastomers do not have a definitive yield point whereas VLDPE and LLDPE materials generally do. As defined in ASTM D-638, yield point is the first point on the stress-strain curve at which an increase in strain occurs without an increase in stress.

In the "Future-Pak '91" publication by Mitsui, the following information is reported on page 314 for yield strengths: Tafmer A-4090, no value reported; VLDPE, 0.896 g/cm<sup>3</sup> density, is 42 kg/cm<sup>2</sup>; and VLDPE, 0.907 g/cm<sup>3</sup> density, is 84 kg/cm<sup>2</sup>. This indicates that plastomers do not have a yield point but instead break when sufficient stress is applied. In contrast, VLDPE materials have definite yield points.

The Mitsui results were qualitatively confirmed in a series of tests in which samples of various resins were prepared

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according to the procedure for thin plastic sheeting outlined in ASTM 882-90. The dimensions of these resin samples tested were 1" wide, 4" long and 7-9 millimeters thick. These samples were tested for yield point and tensile strength following Method A, using a 20"/min. constant rate of separation of the grips initially holding the ends of the specimen 2" apart.

Five samples of each material were tested and the results averaged. The results of these tests are summarized in Table E (Yield Strength).

Table E

Yield Strength	
Type Compound and Manufacturer	Average Yield Strength (psi)
Dow Attane™ XU61512.21 VLDPE (0.901 den.)	1020
Dow Attane™ XU61520.01 VLDPE (0.912 den.)	1329
Union Carbide 1137 VLDPE (0.906 den.)	1121
Union Carbide 1192 VLDPE (0.912 den.)	1323
Mitsui Tafmer A-4085 Plastomer (0.88 den.)	No yield point
Mitsui Tafmer A-1085 Plastomer (0.88 den.)	No yield point

Dow and Exxon report the following tensile strengths at break for their ethylene  $\alpha$ -olefins, as shown in Table F:

Table F

Affinity and Exact Tensile Strengths	
Designation and Type Compound	Value (Mpa)
Exxon 3001 LLDPE (D-882)	48 (MD)
	40 (TD)
Exxon 3201 LLDPE (D-882)	51 (MD)
	40 (TD)
Exact™ 4011 plastomer (D-638)	22
Exact™ 3025 VLDPE (D-882)	56 (MD)
	39 (TD)
Exact™ 3034 VLDPE (D-882)	71.9 (MD)
	57.1 (TD)
Exact™ 3033 VLDPE (D-882)	68 (MD)
	62 (TD)
Exact™ 3027 VLDPE (D-882)	56 (MD)
	36 (TD)
Exact™ 3033 VLDPE (D-882)	90 (MD)
	98 (TD)
Affinity™ PL 1840 VLDPE (D-882)	55 (MD)
	52.7 (TD)
Affinity™ PL 1845 VLDPE (D-882)	45.4 (MD)
	33.4 (TD)
Affinity™ PL 1880 VLDPE (D-882)	49.4 (MD)
	26.2 (TD)

Suitable LLDPE materials for practicing this invention include those manufactured and sold by Dow under the name Dowlex™ (trademark of Dow Chemical Company, USA) and by Exxon under the name Escorne® (registered trademark of Exxon Chemical Company, USA). Suitable VLDPE materials for practicing this invention include certain ethylene  $\alpha$ -olefin polymers manufactured and sold by each of Dow under the names Attane™ and Affinity™ (trademarks of Dow Chemical Company, USA), by Exxon under the name Exact™ (trademark of the Exxon Chemical Company, USA) and by Union Carbide.

Representative VLDPE resins include the ethylene-octene copolymers Dow 4001, 4003, PL 1840, PL 1845 and PL 1880 VLDPEs, the ethylene-butene copolymers Union Carbide 1137, Exxon 3027 and 3025 and the ethylenebutene hexene terpolymers Union Carbide 1192 and Exxon 3033. Representative LLDPEs include the ethylene-octene copolymer Dow 2045 and the ethylene-hexene copolymer Exxon 3001 and 3201.

Suitable ethylene  $\alpha$ -olefin plastomer copolymers include certain of those manufactured and sold by each of Exxon under the name Exact™, Mitsui Petrochemical Industries, Ltd. under the name Tafmer, and Dow under the name Affinity™. For example, suitable resins include Mitsui A-4085, A-4090, A-1085 and A-0585 and Exxon 4011.

Summarizing the foregoing, LLDPE provides the needed machinability properties in the outer layers. If there is less than 25 wt.% LLDPE, the irradiated film does not have adequate slip to be processed through the film wrapping, folding and heat sealing steps when employed as an overwrap for an open food-containing tray. On the other hand, if the LLDPE exceeds 75 wt.%, the film loses its needed amount of biaxial shrink and desired low permanent deformation characteristics. The LLDPE density should not exceed 0.925 g/cm<sup>3</sup> as the material would be too crystalline and a stable bubble could not be maintained during the biorientation step. The LLDPE melt index should be below 3.5 g/10 minutes. Higher values result in a blend which is too fluid to form and maintain a stable bubble. The VLDPE in the outer layer is

also needed for good machinability, i.e., slip properties. Plastomer materials do not provide this characteristic. At least 25 wt.% VLDPE in the outer layer is also needed to provide adequate shrink. 100% LLDPE (ethylene  $\alpha$ -olefin of at least 0.917 g/cm<sup>3</sup> density) does not provide adequate shrink.

In the core layer, the first VLDPE should comprise at least 40 wt.% of the layer as lower concentrations cause bubble instability during biorientation and a low shrink force in the final package. On the other hand, the first VLDPE content in the core layer should not exceed 75 wt.% because the film would be too stiff for complete heat sealing. That is, the film would not be sufficiently flexible for end folding and compression for good interlayer contact in the end flaps for heat sealing. The core layer first VLDPE density should be at least 0.905 g/cm<sup>3</sup> to improve burn through resistance and maintain good bubble stability.

Turning now to the second (and lower density) VLDPE in the core layer, it should comprise at least 10 wt.% of the layer, with the plastomer being at least 15%, to retain the stretch recovery and broad range heat seal characteristics. If the ratio of plastomer to second VLDPE is outside of 0.77 to 1.83, the stretch recovery would not be optimized. The second VLDPE density should be at least 0.900 g/cm<sup>3</sup> to provide optimum stretch recovery heat sealing characteristics.

It should be noted in this respect that the second and lower density VLDPE bridges the gap between the plastomer constituent and the first and higher density VLDPE. Inclusion of this second VLDPE constituent improves stretch recovery and it appears to function as a plasticizer for the other constituents by lowering the blends overall crystallinity and increasing its amorphous content. Higher than 0.905 g/cm<sup>3</sup> density for this second VLDPE constituent undesirably reduces the film's stretch recovery.

The biaxially oriented heat-shrinkable film of this invention may be produced by known techniques such as by coextruding at least the core layer and the first and second outer layers on each side of the core layer to form a primary tube as, for example, described in Canadian Patent 982,923. Alternatively, the composite primary tube may be formed by coating lamination, wherein a first outer tubular layer is extruded and thereafter the core and second outer tubular layers are sequentially coated onto the outer surfaces of the first tubular layer and the core layer. As another alternative, the first outer and core outer layers may themselves be coextruded, and the second outer layer thereafter coated onto the outside surface of the core layer. Coating lamination procedures are described in U.S. Patent 3,741,253. As still another alternative, the at least three layer film may be formed as a sheet by the well-known slot casting procedure.

If the at least three layer film has been prepared as a primary tube or converted from a primary sheet into a tube, it may be biaxially oriented by the well-known two step "double bubble" or trapped process. One such process is described in U.S. Patent 3,456,044. This involves reheating the primary tube and simultaneously stretching the tube in the machine direction ("MD") by operating longitudinally spaced nip rolls at different speeds, and stretching the tube in the transverse direction ("TD") by inflating air inside the tube. Suitable stretch ratios are from 2 to 6 with MD/TD ratios of 3 to 5 preferred.

In the practice of this invention, it is essential to crosslink one or more layers of the film. This may be accomplished by, for example, irradiation using high energy electrons, ultraviolet radiation, x-rays, beta particles and the like. The irradiation source can be any electron beam generator operating in a range of 150 kilovolts to 6 megavolts with a power output capable of supplying the desired dosage. The voltage can be adjusted to appropriate levels. If used as the sole crosslinking method, the irradiation is preferably carried out at dosage between 5 MR and 10 MR, and most preferably between 7.5 MR and 8.5 MR.

In order to reduce the amount of radiation needed for crosslinking, crosslinking enhancers that are well-known in the art can be used. They are added to the blend prior to being formed into a film and include, among others, ethylene glycol dimethacrylate, triallyl cyanurate, divinylbenzene and trimethylol propane triacrylate. Additional suitable materials will be apparent to those skilled in the art.

Crosslinking may also be accomplished chemically through utilization of peroxides, as is well known to those of skill in the art.

Processwise, irradiation can be applied to the entire film or to a single substrate layer such as the first outer layer and prior to biaxial orientation if the primary multilayer film is prepared by coating lamination. This type of irradiative crosslinking is for example described in U.S. Patent 3,741,253. Alternatively, if the film is simultaneously coextruded, it may be preferable to irradiate the entire multilayer film after biaxial orientation as, for example, described in U.S. Patent 4,714,638.

A general discussion of crosslinking can be found at pages 331 to 414 of volume 4 of the Encyclopedia of Polymer Science and Technology, Plastics, Resins, Rubbers, Films published by John Wiley & Sons, Inc.

Unless otherwise noted, the resins utilized in the present invention are generally commercially available in pellet form and may be melt blended or mechanically mixed by well known methods using commercially available equipment including tumblers, mixers or blenders. Also, if desired, well known additives such as processing aids, slip agents, antiblocking agents and pigments, and mixtures thereof may be incorporated into the film by blending prior to extrusion.

The resins and any additives are introduced to an extruder (generally one extruder per layer) where the resins are melt plastified by heating and then transferred to an extrusion or coextrusion die for formation into a tube. Extruder and die temperatures will generally depend upon the particular resin or resin containing mixtures being processed and suit-

able temperature ranges for commercially available resins are generally known in the art, or are provided in technical bulletins made available by resin manufacturers. Processing temperatures may vary depending upon other process parameters chosen. In extrusion of the VLDPE and LLDPE blend for the first and second layers as well as the first and second VLDPE and plastomer blend in the core layer, barrel and die temperatures, for example, may range between 325°F and 450°F. However, depending upon such factors as other resins which may be employed, the manufacturing process used and particular equipment and other process parameters utilized, variations are expected. Actual process parameters including process temperatures will be set by one skilled in the art without undue experimentation.

#### EXAMPLE 1 - PREPARING THE FILMS.

The three layer films were prepared by the trapped or double bubble method as described above. In particular, all three layers were simultaneously coextruded, cooled and then reheated for biaxial orientation.

Various percentages of first VLDPE (Attane™ XU 61520.01), second VLDPE (Exact™ 3027), plastomer (Tafmer A 4085) and LLDPE (Escorene® LL 3201) were blended to form first and second outer layers on opposite sides of the core layer. These were either two component or three component blends. Likewise, various percentages of first VLDPE (Union Carbide type 1192), second VLDPE (Exact™ 3027), LLDPE (Escorene® LL 3201) and plastomer (Tafmer A 4085) were blended so as to form the core layer of various three layer films. These blends were either two or three components. All formulations are given in Table I.

With respect to additive packages, the preferred outer layer package used was 3 wt.% of an antifog and cling additive, Atmer® 8112, manufactured by Imperial Chemical Industries Ltd. and comprising 20% antifog agent in LLDPE of 40 melt index, 3 wt.% of a first slip component, Ampacet 10069, manufactured by Ampacet Corporation and comprising glycerol monostearate concentrate in polyethylene, 1 wt.% of a second slip component, Ampacet 10090, a erucamide concentrate in polyethylene and 1 wt.% of a fluoroelastomer processing aid.

The preferred additive package for the core layer comprised 2% by weight of a fluoroelastomer processing aid concentrate.

In each instance, the dry resin outer layer components were blended by tumble mixing and then fed to a single type extruder and a 60 mm diameter die for coextrusion on either side of the core extruder. Likewise, the dry resin core layer components were blended by tumble mixing and then fed to a single screw extruder and then to a 60 mm diameter die for coextrusion with the outer layers flowing through extrusion passages on either side of the core extruder.

The resins were heat plastified and extruded into a primary tube of about 3.2" diameter having about a 0.010 - 0.015" wall thickness. The extruder barrel and die temperatures were 350°F. The primary tube was cooled to about 60°F and then reheated to about 185 - 195°F for biaxial orientation. The machine direction (MD) stretch ratio was about 4.5 to 1 and the transverse direction (TD) stretch ratio was about 4 to 1. Draw point temperature, bubble cooling rates and stretch ratios were adjusted to maximize bubble stability.

The samples were irradiated with a 175 KeV electron beam to a dose of 8 MR.

In all of the sample embodiments of the invention film set forth in the following Examples, with the exception of the prior art W. R. Grace and Company films, the overall thickness of the film samples was about (15.2 - 21.6 µm) (0.6 - 0.85 mils), comprising a first outer layer of about 15 to about 25% of the total thickness, a core layer of about 50% to about 70% and a second outer layer of about 15 to about 25%. For the prior art Ralph-type film in the sample embodiments, the overall thickness was about the same as above and in the same ratios.

Except for the control sample, SSD-310, all of the samples below were produced as described above.

Sample SSD-310 is the control film, made by Cryovac, a division of W.R. Grace and Company. It is believed to be a 3 or 5 layer coextruded multilayer film. This is the standard film presently used in the poultry packaging process.

Sample #16 film is a nonirradiated Ralph formulation.

Sample #47/16 film is a nonirradiated Ralph-type formulation with LLDPE in the core.

Sample #47/16A is the same formulation as #47/16, but irradiated.

Sample #27A is the same as #47/16A and has stabilizers added to prevent the degradation of slip additives.

Sample #27K is a film of a formulation similar to #47/16A, with a reduced plastomer/second VLDPE ratio.

Sample #28D is an irradiated film of a formulation similar to this invention, and is irradiated.

Sample #29B is an irradiated film of a formulation of this invention.

Sample #29D is an irradiated film of a formulation of this invention.

#### EXAMPLE 2 - THE TRAY SLIDE TEST.

The Tray Slide test was performed on all samples except #16. This test was performed to simulate the kind of forces produced on the Ossid 500 around the front flap roller.

A rectangular #3P styrofoam poultry tray that contained a one pound weight was wrapped around its longitudinal axis with a sheet of film, over the top of the tray with the edges overlapping on the bottom of the tray. At this point, the

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tray was enclosed in a film tube with an open side at either end. Next, the part of the film extending over the tray and forming the two ends of the tube were somewhat flattened, forming flaps, and one flap was folded under the tray.

A clamp with a 500 gram weight was attached to the unfolded flap. The tray was then placed on a flat surface with the bottom edge against a 3/8" diameter stainless steel rod with a surface finish of 8 to 16 grind. The weighted flap was draped over the rod, which was clamped in a notch 3/16" by 3/16" at the end of the flattened surface. The tray was then pushed forward from the back with a spring gauge and the peak force required to push the tray forward was recorded.

Tray slide values of greater than 4.0 lbs. result in the tray hanging up at the first flap roller. A sample with a value of less than 3.2 lbs. does not hang up, and samples with values between 3.2 and 4.0 lbs. give variable results. The results are given in Table I.

The test results show the control is in the acceptable range, as are samples #28D, #29B and #29D of the present invention and the Ralph film #47/16 and #27A. The irradiated Ralph-type films, #47/16A and #27K, give variable to unacceptable results.

TABLE I  
LAB TESTS VERSUS OSSID 500 TRIALS

FORMULATION	SSD-310	#16 O MR	#47/16 O MR	#47/16A 8MR	#27A* 8MR	#27K 8MR	#28D 8MR	#29B 8MR	#29D 8MR
Outer Layers <sup>(a)</sup>									
1st VLDPE (XU-61520.01) - 121		43	64	64	63	70	36	30	36
Plastomer (Tafmer A-4085) - 7/4		32	21	21	22	18	-	-	-
2nd VLDPE (Exact 3027) - 92		25	15	15	15	12	-	-	-
LLDPE (Exxon 3201)		-	-	-	-	-	64	70	64
Core <sup>(a)</sup>									
1st VLDPE (U.C. 1192)		100	60	60	60	60	80	70	70
Plastomer (Tafmer A-4085)		-	-	-	-	-	12	17	17
2nd VLDPE (Exact 3027)		-	-	-	-	-	8	13	13
LLDPE (Exxon 3201)		-	40	40	40	40	-	-	-
Lab Data									
Tray slide-peak force (lbs)	3-3.25	-	2.5-3.25	3.75-5	3.25	3.7	2.65-3.1	2.8	3.2
Film/Film slip (lbs)	1.75-2	-	1.75	-	1.75-2	-	1.5	1.0-1.25	1.5-2
Film/metal rod slip (grams)	120	-	160-180	-	220	-	120-130	120-130	120-130
Slip ratio	15.6	-	10.3	-	8.6	-	12	9.1	14.1
Platen seal range	300-400	235-250	245-280	280-400	-	-	-	-	280-400
Stretch recovery	5 sec.	1 sec.	-	1 sec.	-	-	-	-	1-3 sec.
15 min.	12 sec.	-	-	-	-	-	-	-	3 sec.
70 hr.									
Ossid Data									
Flap fold hang up	OK	OK	OK	(2)	OK	(1)	OK	OK	OK
Flap pull back (%)	0	0	0-5	(3)	95	5-10	0	30	0
Heat seal	OK	(7)	(5)	OK	OK	OK	(4)	OK	OK

\*0.12% Hindered phenol stabilizer added

1. Very high noise/binding
2. Some hang-up on the roll for tucking the first flap
3. Not evaluated due to hang-up
4. Some incomplete seals
5. Narrow seal range
6. Additives comprised 4.5-7.0 wt.% of layer
7. Sporadic burn through-no reproducible seal range
8. Additives comprised 3.0-4.0 wt.% of layer

EXAMPLE 3 - THE FILM/FILM SLIP TEST.

The Film/Film Slip test was performed with all samples except #16, #47/16A and #27K. This test shows that at high values, the package tightness remains after wrapping the package longitudinally, as described in Ex. 2 above, whether or not the longitudinal seam is heat sealed. The film shows that it is tacky enough to stick to itself while the remainder of the packaging is completed.

In this test, film was placed on the sintered metal surface of a metal cart having wheels which were placed along guide rails. The metal cart is 12" long x 4.25" wide and is connected to an apparatus which allows a vacuum to be pulled through the inside of the cart. A vacuum was applied and the film was flattened. A pulley drive motor was connected to the front of the cart. This motor can pull the cart forward along the guide tracks at approximately 10.2 mm/sec (2' per minute).

Another sheet of the same film was wrapped around a brass bar 3" long x 1.5" wide x 0.375" high. This sheet of film was secured with double sided tape to the bar and kept wrinkle free on the bottom of the bar. A spring gauge with the ability to measure up to 5 pounds of tension was attached to the brass bar.

The pulley drive motor was then turned on, pulling the cart along the guide rails. The peak force was recorded for each of the test samples and used in the calculations of slip ratio as shown in Table I.

EXAMPLE 4 - THE FILM/METAL ROD SLIP TEST.

The Film/Metal Rod Slip test was performed on each of the samples described above except for #16, #47/16A and #27K. The purpose of this test was to produce the denominator of the Slip Ratio. It has also been found that films with high number results show a tendency to bind or jam up on metal rollers.

The materials used in this test were a tray slide fixture having an L-shaped shelf cut into one end and having a metal rod placed on this shelf; a 2" x 14" film strip of each of the samples described above; a 1000 gram full scale spring gauge; two binder clamps and a 50 gram weight. Each test was performed as follows:

One clamp was attached to each end of the film strip. The spring gauge was zeroed and attached to one arm of one of the clamps and the 50 gram weight was attached to an arm of the clamp on the opposite end of the film strip. The spring gauge-clamp and a small portion of the film strip were placed on the surface of the tray slide fixture, while the remainder of the film was hung over the metal rod in the end of the tray slide fixture. The spring gauge was then slowly pulled away from the metal rod across the tray slide fixture, drawing the film across the metal rod. The value on the spring gauge while in motion was recorded. Each sample was tested three times with the average number being recorded.

The Slip Ratio was calculated as:

$$\text{Slip ratio} = \frac{\text{Film/Film Slip (lbs)}}{\text{Film to metal rod slip (gms)}} \times 1000 \text{ (1lb=0.454kg)}$$

This ratio is a indication of the tendency of the film to have a flap pull back problem. The results are given in Table I. Preferred film to metal rod slip is 120 grams. Preferred Slip Ratio to prevent flap pull back is greater than 10.

EXAMPLE 5 - THE STRETCH RECOVERY TEST.

A Stretch Recovery test was performed on samples SSD-310, #16, #47/16A and #29D described above. The purpose of this test was to determine the amount of time it takes each film, after it has been used for overwrapping and has been stretched and shrunk, to recover its shape after being indented. This is an important feature of a commercially usable film in the retail food market.

Two sheets 12" in the machine direction by 7.75" in the transverse direction were cut from each film. Each sheet was used to wrap a flat plastic rectangular board 3/16" thick with dimensions similar to the 3P trays, having a tube approximately 2" long and about 1.5" in diameter fitted into the center of the board and extending approximately 1" above the top surface of the board, where the diameter of the tube was parallel with the board surface, laying the sheet on the top side of the board first and folding the sheet down over its length, and using tape to attach the edges of the sheet to the bottom of the board. Next, the corners of the sheet were folded into the bottom of the board, and finally the flaps on either end of the board were folded in. The flaps were also taped to the bottom. The maximum gap between the pieces of tape was 0.5".

The sheets were shrunk around these test boards in a Belco model ST 2108 hot air shrink tunnel at a temperature of 180°F and a belt speed set at a setting of 3.5. The samples were allowed to cool approximately 15 minutes and 70 hours after shrinking. Then the trays were placed in a stretch recovery tester.



The stretch recovery tester contains a spring loaded plunger with an 11/16" diameter carriage bolt head attached to it. The plunger stops are set such that the carriage bolt head stops when the plunger is fully depressed with the upper side of the bolt head 0.5" below the original film surface that covers the tube centered in the tray.

5 The plunger was depressed on the stretch recovery tester until it bottomed out, thereby pressing the bolt head into the sheet overwrapping the food tray, and then the plunger was released. The time it took for the sheet to return to a flat, wrinkle free surface after the plunger was released was measured. Time was measured in seconds, up to 60 seconds.

10 The results are given in Table I. The 15 minute aged control material recovered in 5 seconds and the Ralph-films, #16 and #47/16A recovered in approximately 1 second. The 15 minute aged #29D, a formulation of the present invention that included a processing aid, recovered in 1-3 seconds. The 70 minute aged control film took 12 seconds to recover while #29D recovered in 3 seconds. These results demonstrate that the film of the present invention performed better than the control film in both the 15 minute and 70 hour test.

#### EXAMPLE 6 - HOT BAR AND PLATEN SEALING TESTS.

15 A series of experiments were performed where nonirradiated Ralph-type film, samples #16 and #47/16, irradiated Ralph-type film sample #47/16A, and film sample #29D of the present invention, irradiated and nonirradiated, were tested to determine their acceptable heat sealing ranges in both hot bar and platen systems.

##### 20 a) Platen Sealing Test.

Each of the film samples were tested as follows:

25 A rectangular 3P polystyrene foam tray, approximately 8.5" Long x 6.5" Wide x 1.25" Deep was wrapped with a cut sheet of sample film approximately 18" x 15.5" as described in Ex. 2, except that both flap ends were folded under the tray. A 5 mm x 10" x 10" sheet of Mylar™ polyethylene terephthalate film (trademark of E.I. DuPont de Nemours & Company, USA) was placed on the bottom extending up the sides of the tray. It was taped in place with one piece of tape on two opposite ends of the Mylar™ film.

30 The platen was preheated to the desired temperature. The bottom of the wrapped tray was placed in contact with the platen while having a 6 lb. compression weight on the top of the tray. The tray was left on the platen anywhere from 2 to 8 seconds. At the end of this time, the tray was removed from the platen, placed on the laboratory countertop and rotated rapidly for 10 - 20 seconds to cool the seal. Next, cool tap water was run on the Mylar™ film. The Mylar™ film was then removed and the seal inspected. The results of each sample tested are given in Tables I and J.

##### 35 b) Hot Bar Sealing Test.

The hot bar sealing test determines the acceptable temperature ranges for heat sealing plastic films using a thermal bar heat sealer. The same samples tested in the Platen Sealing Test were tested here. The tests were performed as follows:

40 A Sencorp systems Model 24-AS laboratory sealer manufactured by Sencorp Systems, Inc., Hyannis, Massachusetts, USA, was used. The thermal bar heat sealer is equipped with an upper 1/4" wide sealing bar that may be heated to variably controlled temperatures. In this test, two 1" wide x 4" long (TD direction) samples were cut from the sample films. The sealer was equipped with controls for temperature, time and seal bar pressure. The controls were set at 1.0 seconds dwell time, the time that the upper heated jaw is held against the lower 3/8" thick x 1" wide silicone pad, and at 50 psi jaw pressure.

45 Two film samples were held together and placed between the upper jaw and lower sealing platen of the sealer. The upper jaw and lower pad have glass fiber reinforced release covers to prevent film from sticking to the sealing surfaces. By trial and error, the minimum temperature to seal the two film portions to each other was determined by pressing the jaw against the pad at the prescribed pressure and time with various temperature settings.

50 The maximum temperature was then determined for a similar film sample by placing the adjoining film portions between the covered sealer jaw and pad and closing the upper seal bar down on the lower pad. The film sample was observed after trial and error applications of higher temperatures and the temperature that did not cause a break in the seal, burn through or significant distortion of the seal was determined. The maximum temperature is the last noted temperature obtained before observing a break in the seal integrity.

The results are given in Table J below.

55

Table J

Hot Bar and Platen Seal Ranges <sup>(1)</sup>			
Sample No.	#16	#47/16; 47/16A	#29D
Hot bar seal range (0 MR)	230-280	220-280	260-270
Hot bar seal range (8 MR)	230-460 <sup>(2)</sup>	380-500+	310-500+
% increase seal range	360%	100 + %	1800 + %
Platen seal range (0 MR)	235-250 <sup>(3)</sup>	240-275	250-260
Platen seal range (8 MR)	270-330 <sup>(3)</sup>	280-400+	280-400+
% increase seal range	Burn through unusable	240 + %	1100 + %

<sup>(1)</sup> All temperatures are in °F

<sup>(2)</sup> 5 MR

<sup>(3)</sup> Sporadic burn through

As can be seen from Table J, the effective heat sealing ranges for the Ralph-type film samples #16, #47/16 and #47/16A are quite wide for the hot bar type sealing systems, i.e., at least 50°F. However, they are substantially narrower in a platen-type system, i.e., no more than 35°F.

Table J also shows that for a nonirradiated film having outer layers and core formulations according to this invention, sample #29D nonirradiated, the effective heat sealing ranges for both the hot bar and platen systems were very narrow and impractical, i.e., 10°F, for the platen heat sealing systems.

From this background and from prior experience in films, one skilled in the art might expect that irradiation of these inventive films would have some beneficial effect in terms of widening the extremely narrow heat sealing range of films having the present formulations for the outer layers and core layer and that the sealing ranges of the Ralph-type formulation films would be much broader after irradiation. Surprisingly, after irradiating the present formulation multilayer film sample #29D with 8 MR, the hot bar and platen heat sealing ranges were widened to a much broader extent than expected.

The hot bar seal range for sample #29D irradiated was widened from 260-270°F to 310-500+°F or a 1800+% increase in sealing range, as compared to the 360% and 200+% increases seen in the Ralph-type films. Likewise, platen seal range for sample #29D irradiated was widened from 250-260°F to 280-400+°F, a 1100+% increase in sealing range. Although an increase in the Ralph-type film #16 is reported, it is also seen that sporadic burn through occurred, making the film unusable in commercial applications, whereas the other Ralph-type film, #47/16, showed a 240+% increase in the platen sealing range. In both instances, the film of the present invention showed much larger increases in sealing ranges than did the other films.

#### EXAMPLE 7 - OSSID 500 WRAPPING TESTS.

A series of wrapping tests were performed using the Ossid™ 500 wrapping-platen sealing-platen cooling system.

The standard sized 3P rectangular styrofoam trays as described above were filled with a one pound bag of rice to simulate poultry cuts. The nine sample films used were the same as described in Example 1.

The Ossid™ 500 system was operated at a wrapping rate of about 28 trays per minute. The platen was set at a temperature of about 340 - 350°F, the tray heat contact time was about 2 - 4 seconds, and the tray belt speed was about 50 ft/min. Between about 25 and 50 food packages were wrapped with each film sample.

The rice filled trays were placed on the Ossid™ 500, which automatically overwrapped the trays with film as described above. The trays overwrapped with the nine film samples were then automatically platen heat sealed by the Ossid™ 500. Each of the packages were inspected for machinability, or the suitability of the film to be used on this machine, flap pull back problems and the quality of the heat seals produced on the machine. The results are qualitatively summarized in Table I, which shows that samples of the present invention, #28D and #29D performed as well as the control, SSD-310, while the Ralph-type films, both irradiated and nonirradiated, had various problems with heat sealing, flap pull back and flap hang up on the rollers.

#### EXAMPLE 8 - MACHINABILITY TESTS.

In this series of food-containing tray wrapping tests, the Ossid™ 500 wrapping and heating-platen cooling system

was used to wrap the same type tray used in Example 7, containing one pound bags of rice. Three types of films were used: SSD-310 control and two sample films of the present invention, #29D as described in Ex. 1 and with different amount of additives as noted on Table K. The operating conditions were approximately the same as used in Example 7, but the total number of trays packaged with each type film is reported in Table K. All wrapped trays were examined for various types of tray seal defects, and the results are presented as percent defects of the total number of wrapped trays.

The results are reported in Table K. These results show that the films of the present invention are equivalent to the SSD-310 control film in terms of machinability and sealability.

For purposes of this Example 8, the various types of defects are described as follows:

- a) burn through: any hole or penetration of the film which results from melting/shrinking of the film during sealing which can result in package leakage.
- b) incomplete seal: any package which does not completely seal due to lack of bonding between film layers, forming a potential leaker.
- c) flap pull back: the occurrence of at least one of the end flaps being pulled out from the bottom of the tray during or following the flap folding operation and prior to entering the sealer/cooler.

Table K

Tray Seal Defect Tests <sup>(1)</sup>				
Film Sample No.	No. Trays Run	Burn Through	Incomplete Seal	Flap Pull Back
SSD-310	493	0	0	0.8
#29D	514	0	0.2	0
#29D <sup>(2)</sup>	493	0.4	0	0

<sup>(1)</sup> All results are in percent.

<sup>(2)</sup> Same composition as sample #29D except the core layer contained the 3.0 wt.% additive package.

In summary, the test results from Examples 1-8 show that: a) sample #16 gave a narrow sealing range in the Platen Test; b) sample #47/16 had a broader sealing range than did #16, but not yet broad enough for commercial use; c) sample #47/16A had a very broad sealing range but it jams at the front flap folder on the Ossid™ 500, which jamming correlates with the Tray Slide test value of greater than 4.0 lbs; d) sample #27A, a #47/16-type film, had a moderate level of front flap pull back that was predicted by a decreased Slip Ratio, making it unsuitable for use on the Ossid™ 500, even though it had no front flap fold jamming problems; e) sample #27K had higher Tray Slide values and a high level of flap pull back on the Ossid™ 500; f) sample #28D had incomplete sealing on the Ossid™ 500; g) sample #29B had a low Slip Ratio and a moderate level of front flap pull back was observed, and h) sample #29D showed all the preferred laboratory test properties and performed well on the Ossid™ 500 in all respects.

Further modifications of the invention will be apparent to those skilled in the art, modifications which will be within the scope of the invention as defined in the following claims.

#### Claims

1. A crosslinked biaxially oriented heat shrinkable multilayer stretch film comprising at least a first outer layer, a second outer layer and a core layer between said first and second outer layers, said first and second outer layers each comprising a two component polyethylene blend of between 25 and 75 wt.% very low density polyethylene ("VLDPE") of density between 0.900 and 0.914 g/cm<sup>3</sup>, and between 25 and 75 wt.% linear low density polyethylene ("LLDPE") of melt index below 3.5 g/10 minutes and density between 0.917 and 0.925 g/cm<sup>3</sup>, said LLDPE comprising less than 35 wt.% of the total film weight; and said core layer comprising a three component blend of between 40 and 75 wt.% first VLDPE of density between 0.905 and 0.914 g/cm<sup>3</sup>, between 10 and 35 wt.% second VLDPE of density between 0.900 and 0.905 g/cm<sup>3</sup>, and between 15 and 35 wt.% ethylene  $\alpha$ -olefin plastomer copolymer of density below 0.900 g/cm<sup>3</sup>.
2. A film according to claim 1, which is heat shrinkable and crosslinked such that when subjected to the Platen Test on a plate heated to a contact surface temperature range between 138°C (280°F) and 204°C (400°F) for a contact time of between 2 and 4 seconds, the film heat seals and does not burn through.

3. A film according to any one of claims 1 and 2, wherein in the first and second outer layers the VLDPE comprises from 30 to 40 wt.% and the LLDPE comprises from 60 to 70 wt.%.
4. A film according to any one of claims 1 to 3, wherein in the core layer the first VLDPE comprises from 60 to 75 wt.%, the second VLDPE comprises 13 to 20 wt.% and the plastomer comprises 15 to 25 wt.%.
5. A film according to any one of claims 1 to 4, wherein the ratio of plastomer to second VLDPE in the core layer is 0.77 to 1.83, particularly 1.29 to 1.42.
6. Hermetically sealed and evacuated food package comprising a tray with a bottom section surrounded by upwardly extending side and end walls, perishable food supported on the upper surface of the tray bottom section, and a stretched heat shrunk film extending over each of the food, the side and end wall upper edges and at least part of the lower surface of the tray bottom section and heat sealed to itself in flattened relationship against said lower surface, characterized in that said film is the crosslinked biaxially oriented heat shrinkable multilayer stretch film of any one of claims 1 to 5.
7. Process for platen heat sealing the overlying folded ends of multiple contiguous layers of a stretched thermoplastic film covering a food-containing tray having side and end walls upwardly extending from a bottom section wherein the multiple layers of film are folded over the tray end walls, pressed against the lower surface of the tray bottom section and interlayer heat bonded together to form a hermetically sealed and evacuated food package, characterized by using the crosslinked biaxially oriented heat shrinkable multilayer stretch film of any one of claims 1 to 5; providing a flat metal plate as the platen surface; heating the upper surface of said plate to a temperature between (149 and 204°C) (300 and about 400°F); contacting downwardly and inwardly pressed folded end portions of the stretched film cover with the heated metal plate for a period of between 2 and 4 seconds so as to bond the contiguous film layers of said folded end portions without burn through of said film; and thereafter immediately cooling heat bonded end portions of said film to a temperature below 93°C (200°F).

#### Patentansprüche

1. Vernetzte biaxial orientierte heißschrumpfbare mehrlagige Streckfolie, welche mindestens eine erste Außenschicht, eine zweite Außenschicht und eine Kernschicht zwischen der ersten und zweiten Außenschicht umfaßt, wobei die erste und zweite Außenschicht jede eine Zweikomponenten-Polyethylenmischung aus zwischen 25 und 75 Gew.-% eines Polyethylens mit sehr geringer Dichte ("VLDPE") mit einer Dichte zwischen 0,900 und 0,914 g/cm<sup>3</sup> und zwischen 25 und 75 Gew.-% an linearem Polyethylen mit geringer Dichte ("LLDPE") mit einem Schmelzindex von weniger als 3,5 g/10 Minuten und einer Dichte zwischen 0,917 und 0,925 g/cm<sup>3</sup> umfassen, wobei das LLDPE weniger als 35 Gew.-% des Foliengesamtgewichts ausmacht; und wobei die Kernschicht eine Dreikomponentenmischung aus zwischen 40 und 75 Gew.-% eines ersten VLDPE's mit einer Dichte zwischen 0,905 und 0,914 g/cm<sup>3</sup>, zwischen 10 und 35 Gew.-% eines zweiten VLDPE's mit einer Dichte zwischen 0,900 und 0,905 g/cm<sup>3</sup> und zwischen 15 und 35 Gew.-% eines plastomeren Ethylen- $\alpha$ -Olefin-Copolymeren mit einer Dichte von weniger als 0,900 g/cm<sup>3</sup> umfaßt.
2. Folie gemäß Anspruch 1, welche heißschrumpfbar und vernetzt ist, so daß wenn sie dem Plattentest auf einer Platte, welche auf eine Oberflächenkontakttemperatur im Bereich zwischen 138°C (280°F) und 204°C (400°F) erwärmt ist, während einer Kontaktzeit zwischen 2 und 4 Sekunden unterzogen wird, die Folie heißverschweißt und nicht durchbrennt.
3. Folie gemäß einem der Ansprüche 1 und 2, wobei in der ersten und zweiten Außenschicht das VLDPE von 30 bis 40 Gew.-% umfaßt und das LLDPE von 60 bis 70 Gew.-% umfaßt.
4. Folie gemäß einem der Ansprüche 1 bis 3, wobei in der Kernschicht das erste VLDPE von 60 bis 75 Gew.-% umfaßt, das zweite VLDPE 13 bis 20 Gew.-% umfaßt und das Plastomer 15 bis 25 Gew.-% umfaßt.
5. Folie gemäß einem der Ansprüche 1 bis 4, wobei das Verhältnis von Plastomer zu zweitem VLDPE in der Kernschicht 0,77 bis 1,83, insbesondere 1,29 bis 1,42 beträgt.
6. Hermetisch verschweißte und evakuierte Nahrungsmittelverpackung, umfassend eine Schale mit einem Bodenbereich, welcher von sich nach oben erstreckenden Seiten- und Endwänden umgeben ist, ein auf der oberen Oberfläche des Schalenbodenbereichs liegendes leichtverderbliches Nahrungsmittel und eine gestreckte

heißschrumpfbare Folie, welche sich über jeweils das Nahrungsmittel, die oberen Ränder der Seiten- und Endwände und mindestens einen Teil der unteren Oberfläche des Schalenbodenbereichs erstreckt und mit sich selbst auf eine gegenüber der unteren Oberfläche flachgelegte Weise heißverschweißt ist, dadurch gekennzeichnet, daß die Folie die vernetzte biaxial orientierte heißschrumpfbare mehrlagige Streckfolie aus einem der Ansprüche 1 bis 5 ist.

7. Verfahren zum Platten-Heißverschweißen der übereinanderliegenden gefalteten Enden von angrenzenden Mehrfachschichten einer gestreckten thermoplastischen Folie, welche eine ein Nahrungsmittel enthaltende Schale mit sich von einem Bodenbereich nach oben erstreckenden Seiten- und Endwänden umhüllt, wobei die Mehrfachschichten der Folie über die Schalenendwände gefaltet werden, gegen die untere Oberfläche des Schalenbodenbereichs gepreßt werden und mit sich selbst Zwischenschicht-Heißverbunden werden, um eine hermetisch verschweißte und evakuierte Nahrungsmittelverpackung auszubilden, dadurch gekennzeichnet, daß

die vernetzte biaxial orientierte heißschrumpfbare mehrlagige Streckfolie aus einem der Ansprüche 1 bis 5 verwendet wird;

eine flache Metallplatte als die Plattenoberfläche verwendet wird;

die obere Oberfläche der Platte auf eine Temperatur zwischen 149 und 204°C (300 und ungefähr 400°F) erhitzt wird;

die nach Unten und Innen unter Druck gefalteten Endabschnitte der Streckfolienumhüllung während eines Zeitraums zwischen 2 und 4 Sekunden mit der erhitzten Metallplatte in Kontakt gebracht werden, um die angrenzenden Folienschichten der gefalteten Endabschnitte zu verbinden, ohne daß die Folie durchbrennt; und

anschließend die heißverbundenen Endabschnitte der Folie sofort auf eine Temperatur unterhalb von 93°C (200°F) abgekühlt werden.

## Revendications

1. Film étirable multicouche réticulé à orientation biaxiale, thermorétractable, comprenant au moins une première couche extérieure, une seconde couche extérieure et une couche centrale entre lesdites première et seconde couches extérieures, lesdites première et seconde couches extérieures comprenant chacune un mélange de polyéthylènes à deux constituants, formé de 25 à 75 % en poids d'un polyéthylène très basse densité ("PETBD") ayant une masse volumique de 0,900 à 0,914 g/cm<sup>3</sup>, et de 25 à 75 % en poids d'un polyéthylène linéaire basse densité ("PELBD") ayant un indice de fluidité inférieur à 3,5 g/10 minutes et une masse volumique de 0,917 à 0,925 g/cm<sup>3</sup>, ledit PELBD représentant moins de 35 % en poids du poids total du film ; et ladite couche centrale comprenant un mélange à trois constituants de 40 à 75 % en poids d'un premier PETBD ayant une masse volumique de 0,905 à 0,914 g/cm<sup>3</sup>, 10 à 35 % en poids d'un second PETBD ayant une masse volumique de 0,900 à 0,905 g/cm<sup>3</sup>, et 15 à 35 % en poids d'un plastomère copolymérique éthylène- $\alpha$ -oléfine ayant une masse volumique inférieure à 0,900 g/cm<sup>3</sup>.
2. Film suivant la revendication 1, qui est thermorétractable et réticulé de telle sorte que, lorsqu'il est soumis au test sur plateau sur une plaque chauffée à une température de surface de contact comprise dans l'intervalle de 138°C (280°F) à 204°C (400°F) pendant un temps de contact de 2 à 4 secondes, le film subisse un thermosoudage sans être percé par combustion.
3. Film suivant l'une quelconque des revendications 1 et 2, dans lequel, dans les première et seconde couches extérieures, le PETBD représente 30 à 40 % en poids et le PELBD représente 60 à 70 % en poids.
4. Film suivant l'une quelconque des revendications 1 à 3, dans lequel, dans la couche centrale, le premier PETBD représente 60 à 75 % en poids, le second PETBD représente 13 à 20 % en poids et le plastomère représente 15 à 25 % en poids.
5. Film suivant l'une quelconque des revendications 1 à 4, dans lequel le rapport du plastomère au second PETBD dans la couche centrale est un rapport de 0,77 à 1,83, en particulier de 1,29 à 1,42.
6. Emballage alimentaire soudé hermétiquement et mis sous vide, comprenant un plateau avec une section inférieure entourée par un côté s'étendant vers le haut et des parois terminales, une denrée alimentaire périssable portée par la surface supérieure de la section inférieure du plateau, et un film étiré thermorétracté s'étendant sur chacun des bords supérieurs de la denrée alimentaire, du côté et des parois terminales et sur au moins une partie de la surface

inférieur de la section inférieure du plateau et étant thermosoudé à lui-même à l'état aplati contre ladite surface inférieure, caractérisé en ce que ledit film consiste en le film étirable multicouche réticulé à orientation biaxiale, thermorétractable, suivant l'une quelconque des revendications 1 à 5.

- 5 7. Procédé pour le thermosoudage par plateau des extrémités pliées se recouvrant de couches contiguës multiples d'un film thermoplastique étiré couvrant un plateau contenant une denrée alimentaire, comportant un côté et des parois terminales s'étendant vers le haut à partir d'une section inférieure, dans lequel les couches multiples de film sont pliées sur les parois terminales du plateau, pressées contre la surface inférieure de la section inférieure du plateau et liées à chaud les unes aux autres pour former un emballage alimentaire clos hermétiquement et mis sous vide, caractérisé par l'utilisation du film étirable multicouche réticulé à orientation biaxiale, thermorétractable, 10 suivant l'une quelconque des revendications 1 à 5 ; l'utilisation d'une plaque métallique plate comme surface du plateau ; le chauffage de la surface supérieure de ladite plaque à une température comprise dans l'intervalle de 149 environ à 204°C (300 à environ 400°F) ; la mise en contact vers le bas et vers l'intérieur des portions terminales pliées pressées du revêtement formé par le film étiré avec la plaque métallique chauffée pendant un temps de 2 à 4 secondes de manière à lier les couches de film contiguës desdites portions terminales pliées sans percer par 15 combustion ledit film ; puis, immédiatement, le refroidissement des portions terminales liées à chaud dudit film à une température inférieure à 93°C (200°F).

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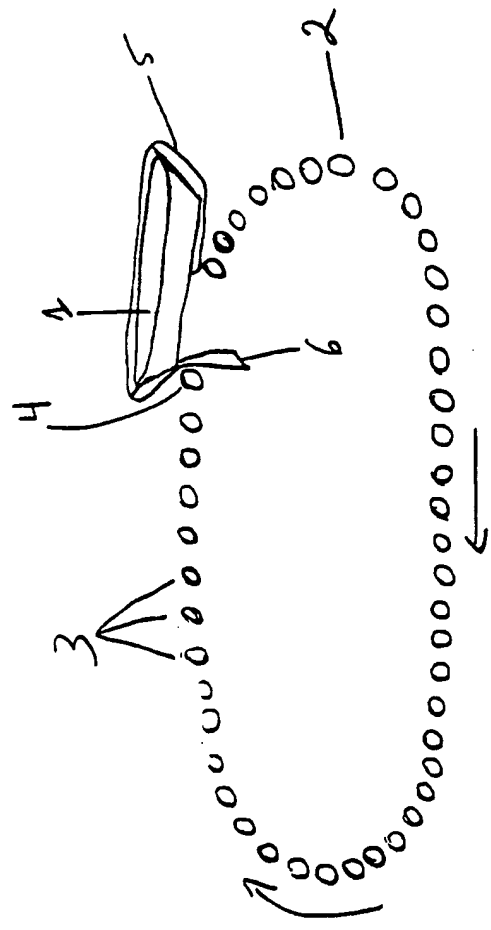


FIGURE 1

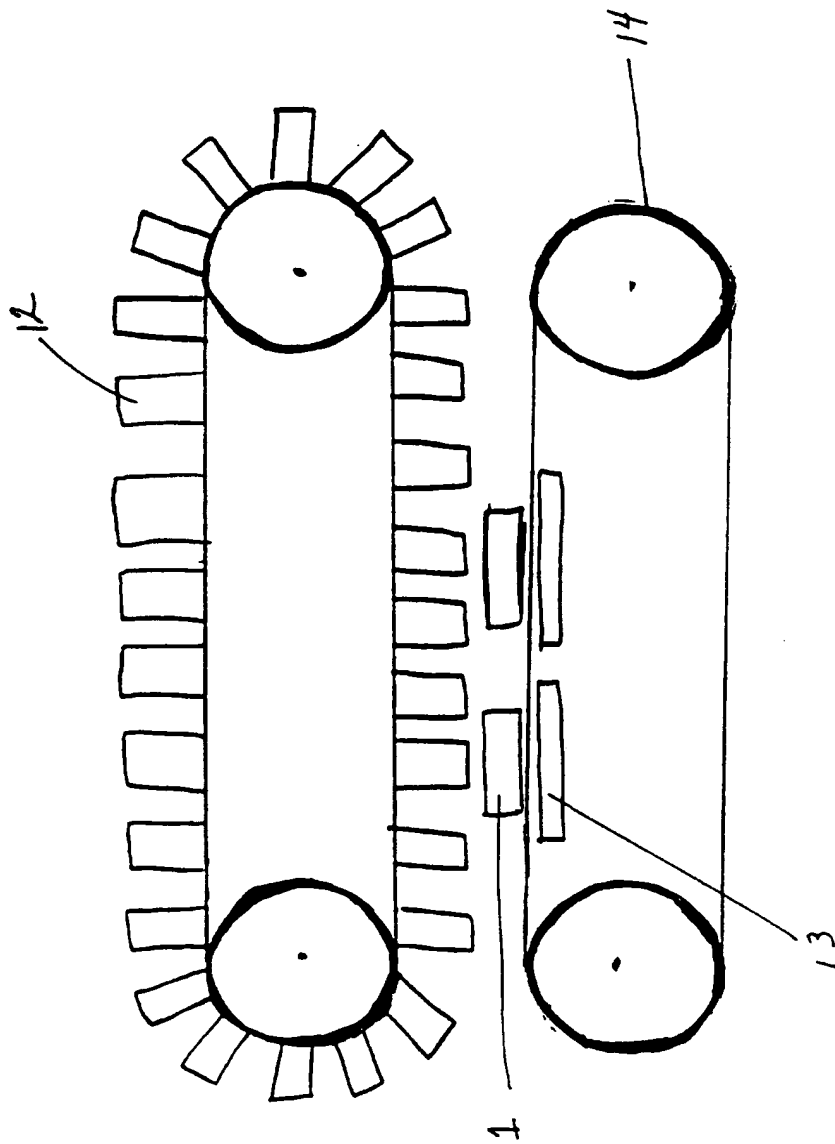


FIGURE 2